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THE EFFECTS OF SMALL DEFORMATION ON CREEP AND STRESS RUPTURE OF--ETC(U)

MAR 82 T E HOWSON, V NARDONE, J K TIEN

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This Air Force sponsored research program aims to enhance understanding of the effects of predeformation on the creep and stress rupture of oxide dispersion strengthened (ODS) alloys and also of the cyclic creep and cyclic stress rupture behavior of ODS alloys. During the third program year, the research effort was focused on the 760°C cyclic creep behavior of MA 6000E, an advanced ODS superalloy that is strengthened by a unique combination of  $\gamma'$  precipitates and oxide dispersoids for both intermediate and elevated temperature strength.

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Cycling the load at 760°C resulted in a dramatic decrease in the minimum strain rate and an increase in the rupture life relative to the static case (no load cycling). The magnitude of the effect increased as the cyclic period decreased --there is an order of magnitude decrease in the minimum creep rate in going from a static test to a cyclic period of ten minutes. The creep deceleration involves storage and recovery of anelastic strain, and this mechanism is discussed and modeled. Comparisons of the cyclic creep properties of ODS alloys and conventional superalloys show that the effects of reduced minimum creep rate and extended rupture life due to cyclic loading during creep are more significant and pronounced in ODS alloys than in other alloys and superalloys that are not dispersion strengthened.

Progress Report  
on  
THE EFFECTS OF SMALL DEFORMATION ON CREEP AND STRESS RUPTURE  
BEHAVIOR OF ODS SUPERALLOYS

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Timothy E. Howson, Vincent Nardone and John K. Tien

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### Abstract

→ This Air Force sponsored research program aims to enhance understanding of the effects of predeformation on the creep and stress rupture of oxide dispersion strengthened (ODS) alloys and also of the cyclic creep and cyclic stress rupture behavior of ODS alloys. During the third program year, the research effort was focused on the 760°C cyclic creep behavior of MA 6000E, an advanced ODS superalloy that is strengthened by a unique combination of  $\gamma'$  precipitates and oxide dispersoids for both intermediate and elevated temperature strength. Cycling the load at 760°C resulted in a dramatic decrease in the minimum strain rate and an increase in the rupture life relative to the static case (no load cycling). The magnitude of the effect increased as the cyclic period decreased--there is an order of magnitude decrease in the minimum creep rate in going from a static test to a cyclic period of ten minutes. The creep deceleration involves storage and recovery of anelastic strain, and this mechanism is discussed and modeled. Comparisons of the cyclic creep properties of ODS alloys and conventional superalloys show that the effects of reduced minimum creep rate and extended rupture life due to cyclic loading during creep are more significant and pronounced in ODS alloys than in other alloys and superalloys that are not dispersion strengthened. ↙

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## Introduction

Advances in turbine engines are paced by the development and understanding of alloys with higher temperature capabilities than the conventional superalloys currently used. The most recent increases in turbine first stage skin temperatures were realized by application of directionally solidified (DS) and monocrystalline nickel-base superalloys. Even higher engine operating temperatures may be attained by harnessing the potential offered by one of several developing technologies. One major materials advance has been the development of oxide dispersion strengthened (ODS) superalloys for critical engine applications such as combustors and first turbine stage vanes and blades.

ODS alloys have been recognized for over two decades as having the potential of being applied at very high temperatures. However, it is only the recent advances in superalloy powder metallurgy processing, in particular mechanical alloying (1), that have finally resulted in the manufacture of ODS alloys of high and consistent quality and of versatile chemistry that have the required mechanical properties and reliability.

To illustrate the high temperature capabilities of two advanced (and recently commercialized) ODS alloys, Inconel alloys MA 754 and MA 6000E, our data showing stresses to result in creep rupture in 100 hours are plotted as a function of temperature for these two alloys and for other cast and wrought nickel-base superalloys in Fig. 1. MA 754 is a Ni-20Cr solid solution alloy strengthened by dispersed yttrium oxide particles comprising about 1 volume percent. Above 1093°C (2000°F) no conventional superalloy can favorably compare with MA 754. MA 6000E is a highly alloyed superalloy, strengthened by both 50 volume percent of  $\gamma'$  precipitates plus about 1.3

volume percent of dispersed yttrium oxide particles. The unusual microstructure of this alloy is shown in Fig. 2. The unique combination of strengthening mechanisms gives MA 6000E creep strength over a wide temperature range, Fig. 1. At intermediate temperatures the  $\gamma'$  precipitation gives MA 6000E strength characteristic of high strength superalloys while the oxide dispersion gives MA 6000E by far the highest elevated temperature strength. MA 6000E is a potential turbine blade material and MA 754 is a turbine guide vane alloy that is already realizing applications in military engines.

In past work at Columbia, we have studied in detail the static creep and stress rupture behavior of MA 754, MA 6000E and other ODS alloys and integrated the results into and extended current theories of creep deformation. This AFOSR-sponsored research program has extended that work to a study of the effects of small deformations on the creep and stress rupture behavior of ODS alloys and also a study of the cyclic creep and cyclic stress rupture properties of these alloys. The work has been centered on the alloys MA 754 and MA 6000E.

Small deformations were implemented by different pretreatments chosen to address situations of both practical and scientific interest. Creep and stress rupture properties and microstructures of MA 754 were first examined after (a) small amounts of relatively high strain rate tensile creep deformation; (b) the introduction of notches; and (c) hot isostatic pressing (HIP). There has been little study of the effect of small predeformations on subsequent properties which is vital since this type of experiment is a possible simulation of mission environment where brief overloading of turbine engine components can occur. No data on the effect of notches on mechanical properties have been published on the ODS alloys of current aerospace interest.



This information is absolutely necessary, since notches and stress concentrations are always present in engineering structures. There is also a need to study the effect on properties of other pretreatments such as hot isostatic pressing (HIP), in part because HIP treatments may potentially be useful as a beneficial thermomechanical treatment for these types of alloys. Our experimental program and analyses of this work have been detailed in the earlier progress reports submitted under this project.

The study and understanding of the cyclic creep and stress rupture, or very low cycle fatigue behavior of ODS alloys, is essential for advanced materials applications. One recent estimate is that 50 to 90% of turbine airfoil failures can be attributed to low cycle fatigue, and this mode of deformation is one of the least understood factors that control engine life (5). There are many situations of low frequency loading and unloading in turbine engine operation, viz., the basic cycles of engine start-up and shutdown, or the thermal strains that occur during temperature variations resulting from rapid changes of power setting.

Our analysis of the literature on the subject, previously reported, found that the systematic work to characterize cyclic creep and stress rupture properties in alloys of engineering interest is scanty and in high temperature ODS alloys is nonexistent. Beyond the obvious practical need for this work, there is, we feel, considerable scientific interest in the creep and stress rupture behavior of ODS alloys under cyclic loading. The ODS alloys have the potential to demonstrate unusual cyclic creep properties because they may store a large amount of anelastic (time dependent, recoverable) strain in the dislocations bowed between oxide particles.

In our last (second year) progress report, we described the modification

of our experimental facilities for cyclic creep and stress rupture testing and reported on the work carried out with MA 754. In the past fifteen months, the focus of our efforts has been the alloy MA 6000E. The cyclic creep and stress rupture behavior of MA 6000E at 760°C will be described, and comparisons drawn to the cyclic creep properties of MA 754 and of a conventional superalloy show that the effects of cyclic loading during creep are more significant and pronounced in ODS alloys than in other alloys and  $\gamma'$  strengthened superalloys that are not dispersion strengthened. The ongoing work that will be completed during the final program year will be detailed.

Progress Report: Cyclic Creep of MA 6000E

Performing cyclic creep tests on the oxide dispersion strengthened (ODS) alloy MA 754 has resulted in a deceleration of the creep rate as evidence by a decrease in the minimum strain rate and increase in the rupture life relative to the static case. The next step was to determine whether this effect would occur for another ODS alloy, MA 6000E, which differs from MA 754 in two important respects. First, MA 6000E is precipitation as well as oxide dispersion strengthened. Second, MA 6000E has an extremely large grain size. Thus, the effect of cycling the load on an alloy which contains two major modes of strengthening will be documented, and since MA 6000E possesses a texture and an extremely large grain size, essentially no grain boundaries are present in the test specimens. As will be discussed, the lack of grain boundaries aids in determining the mechanism which is responsible for the creep deceleration.

All cyclic tests were carried out at the temperature of 760°C, using modified creep machines described in the second year progress report. The load was cycled between a maximum value of 531 MPa and a minimum value of 41 MPa. The maximum load was chosen to give a rupture life of approximately 50 hours under static creep conditions while the minimum load resulted in no detectable creep at the test temperature. The load was applied in the form of a square wave. That is, the time at maximum load equaled the time at minimum load. The cyclic period, defined as twice the hold time, was varied between 20 hours and 10 minutes.

A MINC computer is now being used to collect data from the cyclic and static creep tests. The MINC gives a continuous plot of strain as well as strain rate versus time while the test is still in progress. This continuous feedback of results provides the capability of switching between cyclic and

static testing at specific intervals during a single test. This type of testing is necessary if the mechanism which accounts for the creep deceleration is to be elucidated. Additionally, the MINC provides the resolution to analyze the fine points of the relaxation data on a cycle-by-cycle basis.

The effect of cycling the load is illustrated by the data listed in Table 1. There is a significant decrease in the minimum strain rate and an increase in the rupture life relative to the static case, as the cyclic period is reduced. The tests carried out at cyclic periods of 40 and 10 minutes were interrupted after 200 hours on load (400 hours of total test duration). At this time there was still no evidence that the specimen had gone into tertiary creep. The creep deceleration of MA 6000E is quite substantial. There is an order of magnitude decrease in the minimum strain rate in going from a static test to a cyclic period of 10 minutes.

By taking the ratio of the cyclic to static minimum strain rate, a normalized plot can be obtained. Such normalized plots are shown in Fig. 3 for the 760°C cyclic creep data of MA 6000E, MA 754 and Udimet 700, a precipitation strengthened superalloy which is being studied for comparison purposes. As can be seen, the creep deceleration is more dramatic for MA 6000E than for MA 754 or Udimet 700. The frequency ( $1/\text{cyclic period}$ ) at which the decrease in the minimum strain rate becomes substantial is very dependent upon the alloy. For example, at a frequency of  $0.10 \text{ hours}^{-1}$ , or a cyclic period of 10 hours, MA 6000E has a strain rate equal to half that of the static case, whereas MA 754 and Udimet 700 show no appreciable creep deceleration at this frequency.

The reason for the different frequency dependences among the alloys must have to do with the mechanism which is responsible for the creep deceleration.

This mechanism involves the recovery of anelastic strain (anelastic relaxation) during the time at minimum load. During the period at maximum load, the creep strain is actually composed of two parts, a nonrecoverable strain and an anelastic strain. During the subsequent period at minimum load, the anelastic strain is recovered and therefore does not contribute to the cyclic creep rate. At higher frequencies a greater percentage of the strain accumulated at maximum load goes into the storage of anelastic strain, resulting in a greater deceleration of the creep rate. Thus, the frequency dependence of the creep deceleration of a material is strongly dependent upon the material's ability to store and recover anelastic strain.

One possible explanation to account for the anelastic relaxation during the time at minimum load involves grain boundary sliding. In fact, grain boundary sliding has been proposed as the major mechanism of anelastic relaxation in some metals (6). By observing anelastic relaxation and creep deceleration during cyclic testing of MA 6000E, an alloy which contains essentially no transverse grain boundaries in specimens used in the tests, a possible mechanism has been ruled out. From the results of MA 6000E, it can be concluded that the mechanism of anelastic relaxation involves dislocation motion in the grains of the material.

In keeping with the proposed mechanism of recovering anelastic strain which is responsible for the observed creep deceleration, an equation has been derived to give the functional dependence of the minimum strain rate with frequency. By fitting a single parameter to the data, the magnitude of the effect of cycling the load can be determined for any given alloy. The lines which appear in Fig. 3 are the best fit values of this equation.

After having deduced that the creep deceleration is the result of

anelastic strain recovery during the time at minimum load, the goal of work in the fourth and final program year is to pinpoint what characteristics of an alloy are necessary for creep deceleration to occur. First, cyclic creep tests will be performed on a nickel-20 percent chromium solid solution alloy, which is essentially MA 754 without the oxide dispersoids. Depending on whether or not deceleration is observed for this series of tests, the importance of the oxides in the deceleration process will be indicated. Second, work will be carried out on superalloy single crystals in order further to determine how grain boundaries effect the cyclic creep rate and further to assess  $\gamma'$  strengthened superalloy as opposed to ODS alloy cyclic creep behavior.

After determining which parts of the microstructure are most important to the creep deceleration process, tests which vary the temperature and the minimum load will be performed. Since the recovery of anelastic strain is a thermally assisted process, the effect of varying the temperature should be documented. Also, if the value of the minimum load becomes too high, anelastic relaxation will be prevented from occurring during the minimum load period. The minimum load at which the cyclic creep deceleration effect is lost may be an important material parameter for an alloy.

In addition to finishing the cyclic work as just described, the experiments to characterize the notch rupture of ODS alloys will be completed. Previously the notch rupture behavior of MA 754 at 760°C was reported. Currently the 760°C notch rupture properties of MA 6000E are being obtained, and before the finish of the final year of the program notch testing will be carried out at 1093°C for both MA 6000E and MA 754.

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3. T.E. Howson, D.A. Mervyn and J.K. Tien: Met. Trans. A 11A (1980) 1609.
4. T.E. Howson, J.E. Stulga and J.K. Tien: Met. Trans. A 11A (1980) 1599.
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TABLE I

Cyclic creep and stress rupture data for MA 6000E at 760°C with load cycling between 531 MPa and 41 MPa.

CYCLIC PERIOD	CREEP RATE $\times 10^{-8}/\text{sec}$	RUPTURE LIFE
Static	9.55	44 hrs
20 hrs	7.24	49 hrs
10 hrs	6.31	65 hrs
6 hrs	3.53	94 hrs
4 hrs	2.04	151 hrs
2 hrs	2.19	149 hrs
40 min	1.45	---
10 min	0.81	---



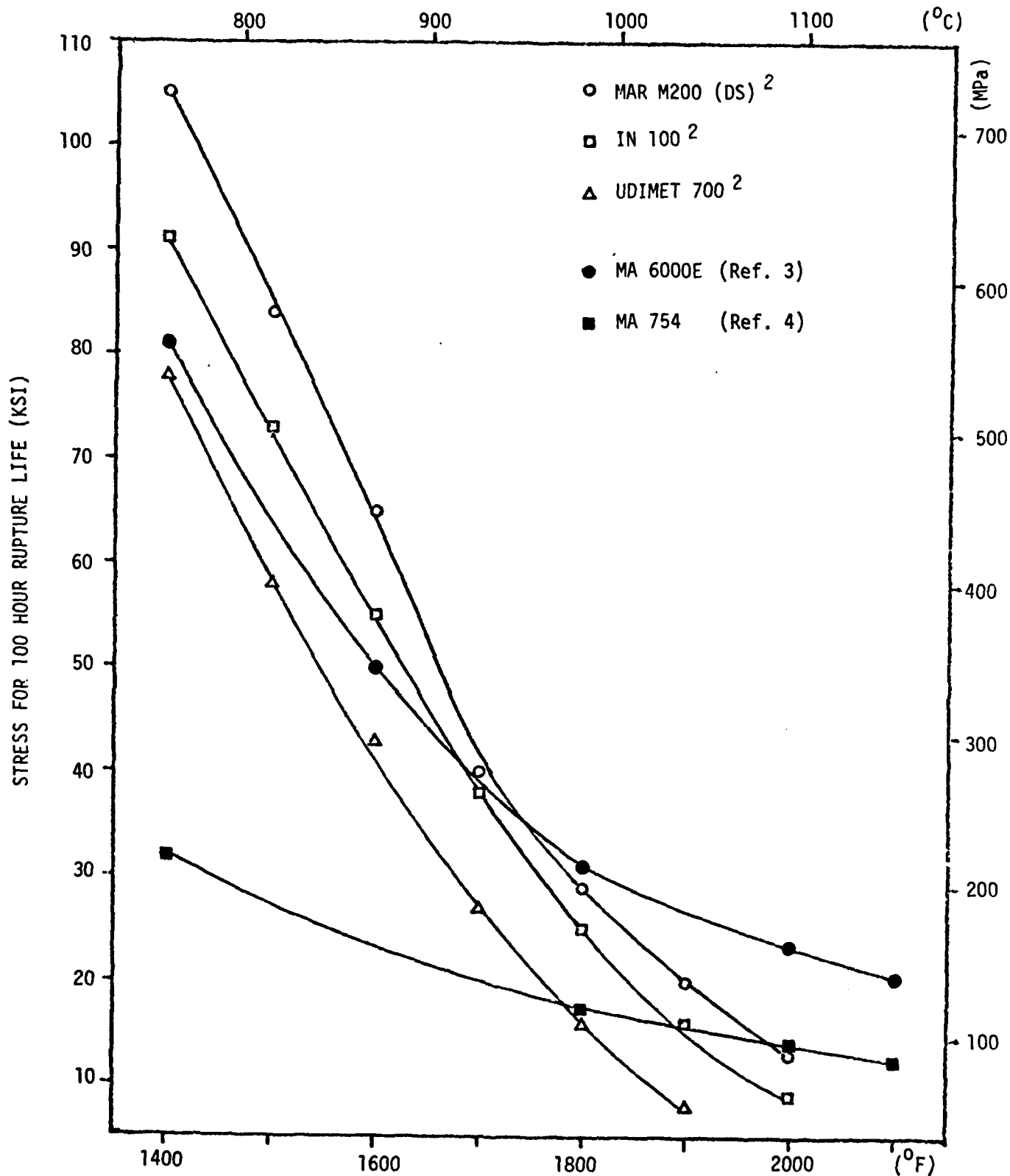


Fig. 1. The stresses to cause creep rupture at 100 hours for MA 754, MA 6000E, and for several conventional cast and wrought nickel base superalloys are compared at different temperatures. The ODS alloys MA 754 and MA 6000E have the best high temperature strength, and MA 6000E also has intermediate temperature strength comparable to the conventional nickel base superalloys.

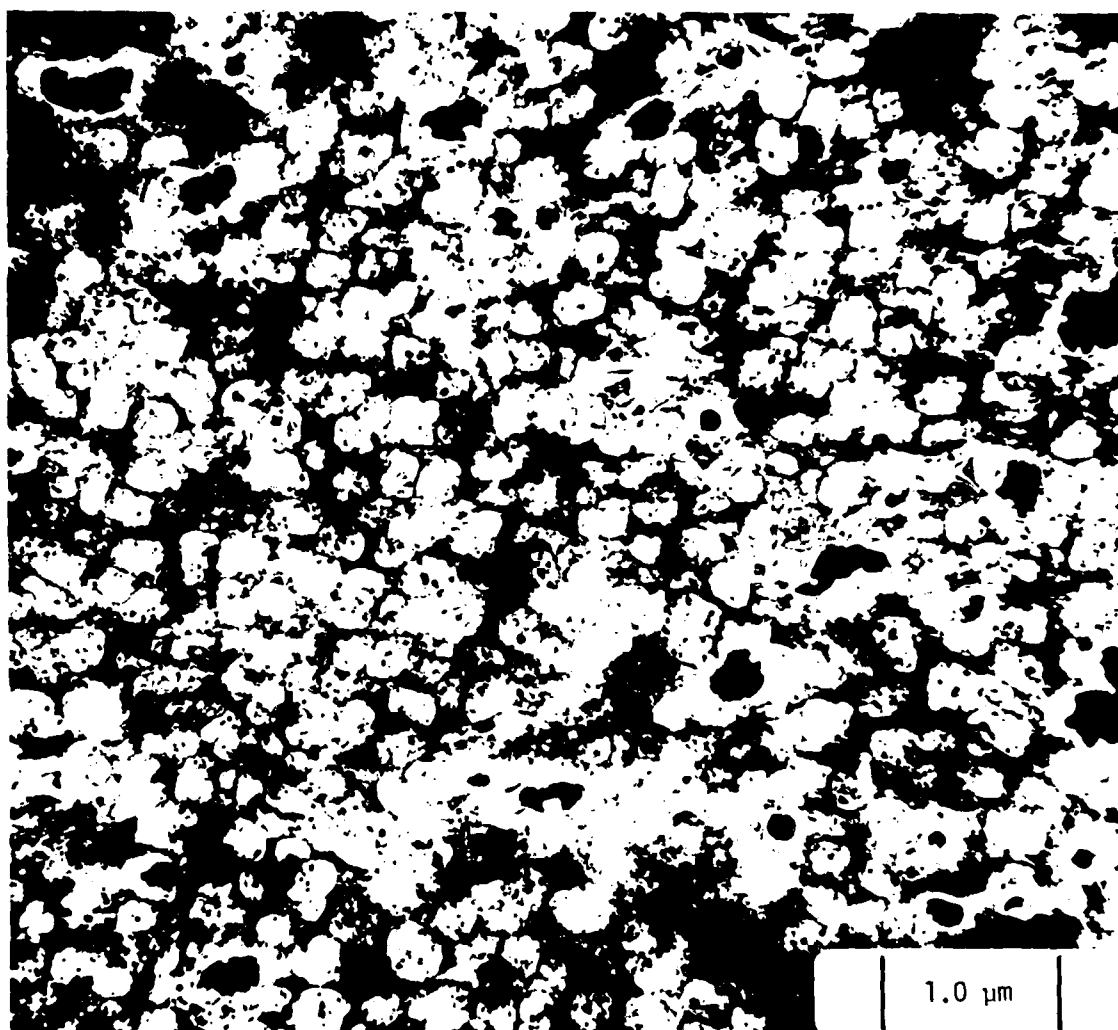


Fig. 2. This dark field transmission electron micrograph shows the unique microstructure of ODS alloy MA 6000E. Both the  $\gamma'$  precipitation and the oxide dispersoids (which are distributed uniformly throughout both the matrix and the  $\gamma'$  phases) can be seen.

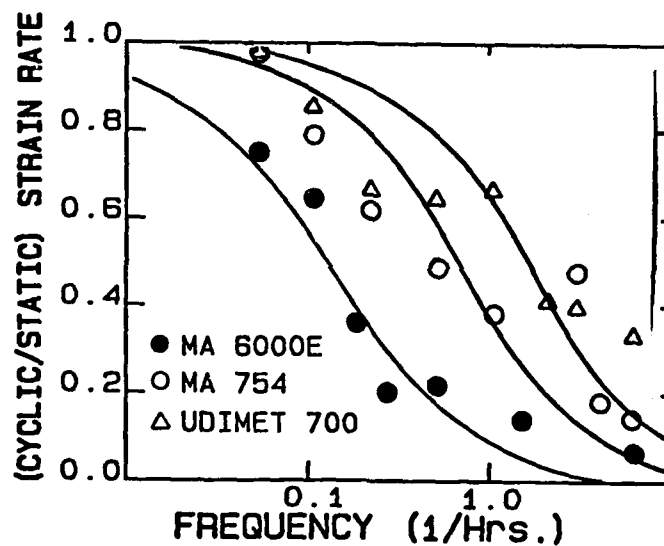


Fig. 3. A normalized plot of the ratio of the cyclic to static minimum creep rate as a function of the cycling frequency is shown for MA 754, MA 6000E and Udimet 700. The cyclic creep deceleration is most pronounced for MA 6000E, and the frequency at which the decrease in the minimum creep rate becomes substantial is alloy dependent, being lower for MA 6000E than for the superalloy Udimet 700 or for the other ODS alloy MA 754.

### List of Publications and Presentations

The following publications and presentations have resulted wholly or in part from the AFOSR grant-funded research.

#### Publications

1. "On the Concept of Back Stress in Particle Strengthened Alloys," S. Purushothaman, O. Ajaja and J.K. Tien, in Strength of Metals and Alloys, Vol. 1, ICSMA5, P. Haasen, V. Gerold and G. Kostorz, eds., Pergamon Press, 1979.
2. "The Role of the Alloy Matrix in the Creep Behavior of Particle-Strengthened Alloys," O. Ajaja, T.E. Howson, S. Purushothaman and J.K. Tien, Mats. Sci. Eng. **44**, 165 (1980).
3. "Creep Deformation and Rupture of Oxide Dispersion Strengthened Inconel MA 754 and MA 6000E," T.E. Howson, F. Cosandey and J.K. Tien, in Superalloys 1980, Proceedings of the Fourth International Symposium on Superalloys, J.K. Tien, S.T. Wlodek, H. Morrow III, M. Gell and G.E. Maurer, eds., ASM Press, 1980.
4. "The Effect of Predeformation on the Creep and Stress Rupture of an Oxide Dispersion Strengthened Mechanical Alloy," R.T. Marlin, F. Cosandey and J.K. Tien, Met. Trans. A **11A**, 1771 (1980).
5. "Anelastic Relaxation, Cyclic Creep and Stress Rupture of  $\gamma'$  and Oxide Dispersion Strengthened Superalloys," J.K. Tien, D.E. Matejczyk, Y. Zhuang and T.E. Howson, Creep and Fracture of Engineering Materials and Structures, B. Wilshire and D.R.J. Owen, eds., Pineridge Press, Swansea, U.K., 1981.
6. "Static and Cyclic Creep and Stress Rupture of Oxide Dispersion Strengthened Nickel-Base Alloys," J.K. Tien, T.E. Howson and D.E. Matejczyk, to be published in Proc. INCO Conf. on Frontiers of High Temperature Materials, New York, May 1981.

7. D.E. Matejczyk, Y. Zhuang and J.K. Tien: "Anelastic Relaxation Controlled Cyclic Creep and Cyclic Stress Rupture Behavior of an Oxide Dispersion Strengthened Alloy," submitted to Met. Trans. A, 1981.

#### Presentations

1. "Effect of HIP Treatment on the Creep Behavior of MA 754," T.E. Howson and J.K. Tien, 108th Annual Meeting of AIME, New Orleans, February 1979.
2. "Notch Sensitive Stress Rupture Behavior of ODS Mechanical Alloy MA 754," F. Nami, D. Matejczyk and J.K. Tien, 108th Annual Meeting of AIME, New Orleans, February 1979.
3. "On the Concept of Back Stress in Particle Strengthened Alloys," T.E. Howson, S. Purushothaman, O. Ajaja and J.K. Tien, Fifth International Conference on the Strength of Metals and Alloys, Aachen, West Germany, August 1979.
4. "Creep and Stress Rupture of ODS and  $\gamma'$  Strengthened Engineering Alloys Under Cyclic Loading," D.E. Matejczyk, T.E. Howson and J.K. Tien, 109th Annual Meeting of AIME, Las Vegas, February 1980.
5. "Predeformation, Localized Deformation, and HIP Effects on Defects and Properties in MA 754," T.E. Howson, F. Cosandey and J.K. Tien, 109th Annual Meeting of AIME, Las Vegas, February 1980.
6. "Effects of Hot Isostatic Pressing on the Structure of an Oxide Dispersion Strengthened Alloy," A. Koren, F. Cosandey, T.K. Glasgow and J.K. Tien, 109th Annual Meeting of AIME, Las Vegas, February 1980.
7. "Effects of Higher Strain Rate Deformation on Structure and Creep Properties of an ODS Alloy," R.T. Marlin, F. Cosandey and J.K. Tien, 109th Annual Meeting of AIME, Las Vegas, February 1980.
8. "Creep and Stress Rupture Behavior of a Nickel-Base Superalloy Under Cyclic Loading," D.E. Matejczyk, Y. Zhuang and J.K. Tien, TMS-AIME Fall Meeting, October 1980.

9. "Cyclic Creep and Stress Rupture Behavior of Nickel-Base ODS and  $\gamma'$  Strengthened Alloys," D.E. Matejczyk, Y. Zhuang and J.K. Tien, 110th Annual Meeting of AIME, Chicago, February 1981.
10. "Notch Sensitive Stress Rupture Behavior of an Oxide Dispersion Strengthened Superalloy," T.E. Howson and J.K. Tien, 110th Annual Meeting of AIME, Chicago, February 1981.
11. "Creep Fracture Processes in ODS Alloy MA 754," C.T. Yen, F. Cosandey, T.E. Howson and J.K. Tien, 110th Annual Meeting of AIME, Chicago, February 1981.
12. "Advances in P/M and ODS Superalloys," J.K. Tien and T.E. Howson, Invited Speaker, 1981 ASM Materials Science Seminar on Advances in Powder Technology, Louisville, KY, October 1981.
13. "Cyclic Creep and Stress Rupture of a Mechanically Alloyed Oxide Dispersion and Precipitation Strengthened Nickel-Base Superalloy," V.C. Nardone, D.E. Matejczyk and J.K. Tien, 111th Annual AIME Meeting, Dallas, February 1982.

Personnel Associated With the Research Effort

Currently Associated

Professor John K. Tien, Principle Investigator

Tim Howson, Post-Doctoral Research Associate (supported by this grant)

Dan Matejczyk, Graduate Research Assistant (supported by this grant)

Vince Nardone, Graduate Research Assistant (independently supported)

Also Contributed to the Research in the Past Year

Frederic Cosandey, Post-Doctoral Research Associate

Zhuang Yi, Visiting Scholar

Yen Chin-Tang, Graduate Research Assistant

Mona McAlarney, Graduate Research Assistant

Charles Burger, Graduate Research Assistant

Alice Cooper, Graduate Research Assistant

Eric Karten, Undergraduate Lab Assistant

Theses Arising from the Research

Fereydoon Nami, "Notch Sensitive Stress Rupture Behavior of ODS Mechanical Alloy MA 754," Master's Thesis, Columbia University, May 1979.

Avi Koren, "The Effect of HIP on the Creep Rupture Properties and the Microstructure of ODS Alloy MA 754," Master's Thesis, Columbia University, October 1979.

Robert Marlin, "The Effect of Predeformation on the Creep and Stress Rupture of an Oxide Dispersion Strengthened Mechanical Alloy," Master's Thesis, Columbia University, October 1979.

Yen Chin-Tang, "Mechanisms of Stress Rupture in the ODS Alloy MA 754," Master's Thesis, Columbia University, September 1981.

Mona McAlarney, "Creep and Stress Rupture of Oxide Dispersion Strengthened Alloys and Superalloys," Master's Thesis, Columbia University, November 1981.

Dan Matejczyk, "Elevated Temperature Cyclic Creep and Cyclic Stress Rupture of Particle Strengthened Alloys," Engineering Science Doctoral Thesis, Columbia University, expected 1982.

Vince Nardone, "Cyclic Creep and Stress Rupture of an ODS Superalloy," Master's Thesis, Columbia University, expected 1982.



### Interactions with Outside Research Groups

1. Several meetings with INCO research personnel at their request to discuss our results.
2. Several meetings with Special Metals Corporation personnel at their request to discuss our results. (Special Metals and INCO are the world's two major producers of ODS superalloys.)
3. Two separate day-long meetings with Prof. Che-Yu Li of Cornell University to present our experimental results and discuss cyclic creep and low cycle fatigue behavior in relation to Cornell's phenomenological models for transient deformation.
4. Seminar at Olin Corporation, New Haven, CT, to present our results and interpretation of cyclic creep behavior for a wide range of alloys.

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